LIITE I

POPE
Itä-Suomen Yliopisto – pöytäkirja 20.11.2012

ITÄ-SUOMEN
YLIOPISTO

22.11.2012

Polttoperäisten päästöjen ja nanohiukkasten haitallisuuden määrittäminen uudella tutkimusmenetelmällä (POPE)
Rahoituspäätöksen numero 40469/10
Diaarinumero 2175/31/2010

Johtoryhmän 4. kokous

Aika:	Tiistaina 20.11.2012 klo 14.00 – 15.00
Paikka:	Itä-Suomen yliopisto, Kuopion kampus, Melania 220 kokoushuone
Läsnäolijat:	Maija-Riitta Hirvonen, UEF Jorma Jokiniemi, UEF Kari Lehtinen, Ilmatieteen laitos Pekka Matilainen, Ecocat Oy Kati Takala, Energiateolliuus Timo-Pekka Veijonen, Stora Enso / Soodakattilayhdistys Stefanie Kasurinen, UEF Kari Kuuspalo, UEF Ari Leskinen, Ilmatieteen laitos Mikko Happo, UEF Karin Koivisto, UEF(sihteeri)

1. **Kokouksen avaus**

   - Maija-Riitta Hirvonen avasi kokouksen klo 14.00.

2. **Puheenjohtajan ja sihteerin valinta**

   - Puheenjohtajaksi ehdotettiin Maija-Riitta Hirvosta ja sihteeriksi Karin Koivistoa. Puheenjohtajan ja sihteerin valinta hyväksyttiin.
   - Kokoukseen osallistujat esittäytyivät.

3. **Esityslistan hyväksyminen**

   - Esityslista hyväksyttiin työjärjestykseksi.

4. **Edellisen kokouksen pöytäkirjan hyväksyminen**

   - Edellisen kokouksen pöytäkirjan hyväksyminen: ei muutoksia, pöytäkirja hyväksyttiin.

5. **Projektin tilannekatsaus:**

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- Jorma Jokiniemi esitteli projektin taustat ja tavoitteet ('Chain of events from heater emissions to health impacts"). Polttoperäisten päästöjen ja nanohiukkasten haitallisuuden määrittämisessä käytetään tutkimusympäristöä, joka mahdollistaa sekä tuoreiden että ikääntyneiden päästöjen haitallisuuden selvittämisen solukokein todellista altistumista vastaavissa olosuhteissa. Käytiin läpi eri päästötilanteet, joita projektissa on tarkoitus tutkia. Projektissa on edetty suunnitelman mukaisesti.

- Maija-Riitta Hirvosen ryhmä tutkii mekanismeja eri terveyshaittojen taustalla. Tunnetaan neljä päämekanismia, jotka osallistuvat terveyshaittojen muodostumiseen: DNA-vauriot, solukuolema, oksidatiivinen stressi ja tulehdus. Projektissa selvitetään hiukkasten osuutta niihin ja selvitetään mahdollista syy-seuraussuhdetta.

a) Soodakattila

- Kari Kuuspalo kertoi, että viikolla 48 kerätään DGI-impaktorilla soodakattilan piipun päästöstä hiukkasmassaa, josta tehdään kemialliset ja toksikologiset analyysit. Mitattavia parametreja ovat mm. hiukkasmassan pitoisuus ja lentotuhka (K, Cd, Ca).
- Soodakattilan osalta hiukkasten keräyksiä tehtiin syyskuun 2012 alusta vanhalla menetelmällä, mutta saatu hiukkasmassa oli riittämätön tarvittaviin mittauksiin (24 mg kerätty, kun tarve olisi ollut 40 mg). Riittävän massan keräämiseksi sekä toksikologiaan että kemiallisiin analyyseihin näytteenottoja jatketaan viikolla 48, ja suunnitellut analyysit suoritetaan mahdollisimman pian.

b) Hakelaitos

- Kari Kuuspalo kertoi että hakelaitoksen mittaukset tehdään vasta talven aikana, jotta kuormat vastaavat todellista tilannetta lämmityskaudella ja kattilan toimiessa normaaliteholla.
- On toivottu, että laitoksessa käytettäisiin tasalaatuisia pellettejä näytteiden keruun aikana, jotta saataisiin kerätyksi mahdollisimman tasalaatuinen näyte.

c) ILMARI:n tilanne

- Stefanie Kasurinen esitteli hopeananohiukkasilla tehtyjen solualtistuskokeiden tuloksia, jotka hän on esittänyt AAAR konferenssissa Minneapolisissa tänä syksynä ('Evaluation of the Toxicological Potential of Silver Nanoparticles').
- Kokeissa käytettiin "air-liquid interface" menetelmää, jonka tarkoitus on jäljitellä *in vivo* olosuhteita mahdollisimman tarkasti.
- *Air-liquid interface:* solut kasvatetaan inserteillä, joissa ne ovat suoraan kosketuksissa tutkittavan näytteen kanssa, mukaan lukien kaasut.
- Muistuttaa todellisia altistusolosuhteita, mutta on teknisesti haastava.
- Alkuperäisen Vitrocell-yksikköön tehtyjen muutosten jälkeen onnistuttiin saamaan parempi hiukkasdepositio.

- Hopeahiukkaset lisäsivät etenkin IL-8 ja IL-6 tuotantoa, muiden mitattujen sytokiinien kohdalla vastetta ei ollut tai se oli selvästi alhaisempi.

- Tulevaisuudessa tulisi nanopartikkelidepositioiden tehokkuutta ja tasalaatuisuutta kehittää, sekä tuottaa malleja joissa käytetään useampaa kuin yhtä solutyyppiä, jotta voidaan jäljitteellä paremmin keuhkoissa vallitsevia olosuhteita.
- Pasi Jalava on tällä hetkellä Saksassa, Hannoverissa, Fraunhofer- Instituutissa, missä hän työskentelee edellä mainitun kaltaisten solumallien kanssa. Myöhemmin tätä tietoa voidaan soveltaa myös Hirvosen ryhmän tutkimuksissa.
- Maija-Riitta Hirvosen mielestä depositiota täytyy vielä parantaa ja kosteutta pitää olla enemmän solualtistuksen aikana.
- Maija-Riitta Hirvonen kertoi, että aerosolien ja päästöjen altistusyksikkö projekti on aloitettu. Tässä EU:n rakennerahaston rahoittamassa projektissa rakennetaan koe-eläinten altistusyksikkö, joka mahdollistaa solu- ja eläinaltistusten tekemisen samanaikaisesti.

6. Talousasiat

- Karin Koivisto esitteli projektin tämän hetkisen rahatilanteen UEF:n osalta.
- Ari Leskinen esitteli projektin rahatilanteen FMI:n osalta.

7. Julkaisut

- Kansainvälisesti referoituja julkaisuja ei ole vielä julkaistu, mutta tuloksista on raportoitu mm. EAC 2012 kokouksessa Granadassa Espanhassa ja AAAR 2012 kokouksessa Minneapolisissa USAssa. Seuraavaksi tuloksia esitellään SOT 2013 kokouksessa San Antoniossa Texasissa USAssa. Jatkossa tuloksista raportoidaan kansainvälisissä kokouksissa sekä referoiduissa julkaisuissa.
- Seuraava EAC Konferenssi (European Aerosol Conference) järjestetään Prahassa, 1.-6.9.2013.
- Stefanie Kasurinen tekee väitöskirjan projektin puitteissa (Maija-Riitta Hirvosen ryhmän jäsen).
- Jorma Jokiniemi mainitsi, että myös hänen ryhmänsä käyttää projektiin liittyviä tieteellisiä julkaisuja väitöskirjojen osatöinä.
- Soodakattilasta ja hakelaitokselta otetut näytteet ja niiden analyysit julkaistaan.

8. Muut asiat

- Kaikki johtoryhmän jäsenet eivät ole vielä nähneet ILMARI tutkimuslaboratoriota. Siksi päätettiin tehdä vierailukierros heti tämän kokouksen jälkeen.

9. Seuraava kokous

- Torstaina 13.6.2013, klo. 13.00 - 15.00, Itä-Suomen yliopisto, Kuopion kampus, Melania 220 kokoushuone. Ari Leskinen lähettää kutsut.
- Puheenjohtaja päätti kokouksen klo. 15.00

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22.11.2012

LIITTEET: 1. Kokouksen agenda 20.11.2012
2. Johtoryhmän osallistujalista 20.11.2012

LIITE 1

Polttoperäisten päästöjen ja nanohiukkasten haitallisuuden määrittäminen uudella tutkimusmenetelmällä, POPE

Johtoryhmän 4. Kokous
Melania 220, Kuopion Kampus 20.11.2012
klo. 14.00–16.00.

Esityslista

1. Kokouksen avaaminen
2. Puheenjohtajan ja sihteerin valinta
3. Esityslistan hyväksyminen
4. Edellisen kokouksen pöytäkirjan hyväksyminen
5. Projektin tilannekatsaus
    a) Soodakattila
    b) Hakelaitos
    c) ILMARI:n tilanne
6. Talousasiat
7. Julkaisut
8. Muut asiat
9. Seuraavasta kokouksesta sopiminen

LIITE 2

POPE osallistujat **20.11.2012, klo 14.00, Melania 220**			
	kyllä	ei	allekirjoitus
Tarmo Hattunen		x	
Maija-Riitta Hirvonen	x		
Jorma Jokiniemi	x		
Jens Kohlmann			
Juha Kouki		x	
Kari Lehtinen	x		
Pekka Matilainen	x		
Markus Nieminen			
Annamari Soikkeli		x	
Kati Takala	x		
Juha Timonen		x	
Timo-Pekka Veijonen	x		
Stefanie Kasurinen	x		
Ari Leskinen	x		
Pasi Jalava		x	
Olli Sippula		x	
Jarkko Tissari	x	x	
Karin Koivisto	x		
Kari Kuuspalo	x		
Mikko Happo	x		

LIITE II

Method for processing of alkaline condensate emanating from the manufacture of cellulose pulp – patentti 20.1.1999

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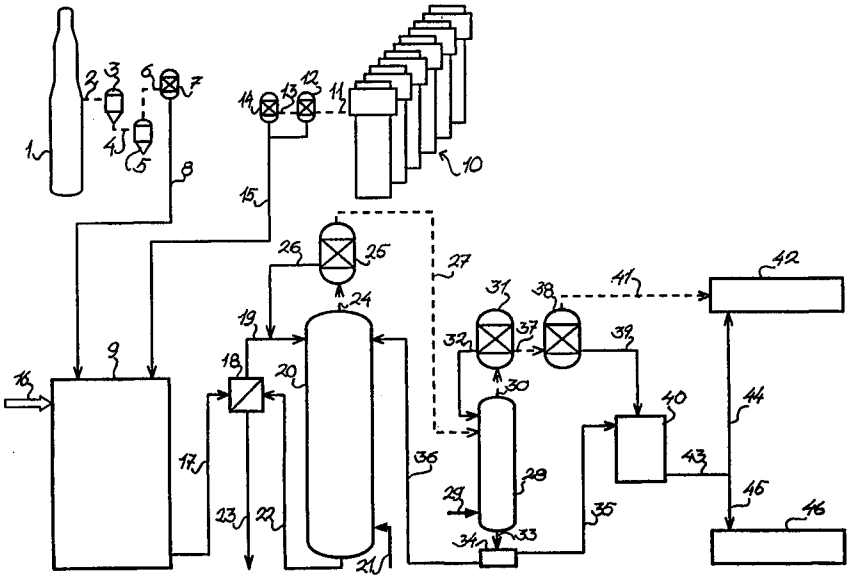

(57) Abstract

The present invention solves the problem concerning the emission of considerable quantities of nitrogen oxides in the combustion of gases generated from stripper gases and/or generated methanol, and relates to a method of processing condensate generated in the manufacture of cellulose pulp by alkaline digestion. The invention is characterized in that acid is added to the condensate prior to stripper treatment, so as to lower the pH of the condensate to ≤7.5 leading to retainment of the major part of the original nitrogen content of the condensate, and thereafter recovering stripper gases generated by the stripper treatment in a conventional manner, and passing cleansed, nitrogen rich condensate to a consumer station.

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Method for processing of alkaline condensate emanating from the manufacture of cellulose pulp

Technical field

The present invention is suitable and will be used in conjunction with the manufacture of cellulose pulp where lignocellulosic material, for instance different types of wood, is digested with the aid of an alkaline cooking liquor. A number of alkaline digestion (cooking) methods are known to the art. Examples of such known methods are the sulphate process, the polysulphide process and processes of the soda type (sodium hydroxide) process, which use catalysts, such as a quinone compound. The term sulphate process also includes, for instance, the application of high sulfidity, the use of counter-current cooking where white liquor is also introduced into the system during an advanced stage of the cook, and chemical treatment of the lignocellulosic material prior to the actual sulphate cook.

The lignocellulosic material is digested under pressure leading to that after the digestion waste liquor and a gaseous mixture consisting of water vapour (steam) and diverse organic and inorganic chemical compounds departing from the waste liquor are caused to leave the digester. In the case of typical batch digestion processes, the waste liquor leaves the digester together with the resultant pulp. The mentioned gas mixture is caused to condense to form a so called digester house condensate. Such gas mixtures are emitted and collected also at locations other than the actual digester in the digester house and caused to condensate, and

hence the name digester house condensate instead of solely blow steam condensate. This condensate is formed in both batchwise and continuous digestion of lignocellulosic material. When digestion of the lignocellulosic material has been completed, the cooking waste liquor, normally referred to as black liquor (now most often in mixture with bleaching waste liquor) is extracted from the resultant cellulosic pulp and evaporated prior to combustion of the black liquor in the form of thick waste liquor, for instance in a recovery boiler. Gas mixtures of kinds similar to the aforedescribed gas mixture are also caused to leave the black liquor in the process of evaporating said liquor. These gas mixtures are caused to condense and therewith form a so called evaporation condensate.

These condensates must be taken care of, either individually or in mixture, and the present invention describes a method of processing said condensates.

Background art

The lignocellulosic material, such as wood for instance, contains a plurality of chemical compounds, including inorganic compounds, such as diverse nitrogen compounds. After the alkaline digestion of said material, the major part of the nitrogen compounds remains in the cooking liquor, probably as dissolved ammonia. Since as well the cooking liquor as the cooking waste liquor are strongly alkaline, the gases from the digester house as well as the gases of evaporation will contain considerable quantities of ammonia. Other volatile substances included in the gas mixtures are, for instance, methanol, diverse terpenes and sulphur compounds. The gas mixtures will, of course also contain water vapour in addition to said substances.

As mentioned earlier, the digester house vapour and the evaporation vapour will both undergo condensation and the resultant condensate till consequently contain said chemicals, which is why the condensate is said to be

contaminated. Condensation of the vapour is effected by bringing the comparatively hot vapour into contact with a chilled surface in a condenser. An alternative term for a condenser is a heat exchanger, this heat exchanger normally being a tube heat exchanger or a spiral heat exchanger. Direct condensation with the aid of cold water can also be employed.

The collected contaminated condensate, which normally consists in a mixture of digester house condensate and evaporation condensate, has a pH value above 8, and often above 9.

The mentioned collected contaminated condensate is passed to a vapour stripper in which the major part of said contaminants are expelled from the liquid in a gaseous state. This process is designated stripping and comprises heating the condensate with steam in a column. The steam may be fresh steam or vapour obtained from liquor in any stage in the liquor evaporation plant. A typical result is that over 90% of the methanol and the sulphur compounds in the original, impure condensate will be removed. Practically all of the nitrogen present in the impure condensate will also be driven off, probably essentially as ammonia. The expelled compounds in the form of so called stripper gases can either be conducted directly to destruction and/or combustion, or can be caused to condense. At this stage, the condensate will consist to a large extent of methanol, which, however, is contaminated to some extent by other components of the expelled gas. This methanol is then used as fuel in an incinerator for odorous gases and/or in a lime kiln and/or in a recovery boiler or in some other boiler.

When stripper gases or methanol obtained in the aforesaid manner are combusted, a significant part of the nitrogen content (probably mainly ammonia), often in the order of magnitude of 40%, is converted to oxides of nitrogen (NO_x). The release of nitrogen oxides is negative from an

environmental aspect, since they have both an acidifying and a fertilising effect on the surroundings.

The cleansed condensate has a very large liquid volume and is normally distributed to different positions in the pulp mill. Part of the condensate is ususally conveyed to the chemical recovery plant, while another part is ususally used to wash the cellulose pulp, while the remainder of the condensate is passed to a drain.

Disclosure of the invention

Technical problem

There is a general desire to restrict the emission of nitrogen oxides to atmosphere to the greatest possible extent, since such emission is negative from an environmental point of view of earlier described grounds. For this reason, the emission of nitrogen oxides is taxed fiscally in certain countries, therewith adding to the cost of the cellulose pulp manufacturing process.

The solution

The present invention contributes towards a solution of the aforesaid problem and relates to a method of processing condensate deriving from the manufacture of cellulose pulp by means of alkaline digestion, said method being characterized by introducting acid into the condensate prior to stripper treatment, such as to lower the pH of the condensate to ≤7,5 leading to retainment of the major part of the original nitrogen content of the condensate, and by recovering stripper gases after the stripper treatment in a conventional manner and passing cleansed, nitrogen rich condensate to a consumer site.

By the expression major part is meant preferably that at least 75% of the original nitrogen content of the condensate still remains in the condensate after the stripper treatment.

By the expression nitrogen rich is not meant that the cleansed condensate contains a high total amount of nitrogen, but merely that the amount of nitrogen present is high in comparison with cleansed condensate that has been processed in a conventional manner, where the nitrogen content is very low and sometimes almost zero.

The contaminated or original condensate, both in the form of digester house condensate and evaporation condensate and a mixture of these condensates, has a pH value above 8, normally above 9.

The amount of acid introduced into the system will preferably be sufficient to lower the pH of the contaminated condensate to ≤6.5. Any acid whatsoever may be used. A suitable acid in this respect is sulphuric acid.

According to the invention, it is fully possible to treat digester house condensate and evaporation condensate separately in the afordescribed manner. However, it is preferrred to first mix these condensates together and then treat the mixture in accordance with the invention.

The acid can be delivered to the two streams of condensate, either individually or subsequent to mixing said streams. The acid may also be delivered in the mixing tank or at some location downstream of said tank, although upstream of the stripper column. The acid may, of course, be delivered to solely one of the condensate streams in a larger amount sufficient to ensure that the appropriate pH value will be obtained when the two streams are mixed together.

As far as can be judged, lowering of the pH of the condensate in accordance with the invention will result in the ammonia dissolved in the condensate being converted successively to ammonium ions (NH_4^+), wherewith a predominant part of said ions will remain in the condensate in the stripping process.

All of the cleansed nitrogen rich condensate, or a part of said condensate, can be delivered to any chosen consumer station. It is preferred to deliver the condensate

to a place where nitrogen is required, for instance to a biological effluent or sewage purification plant within the sulphate pulp mill concerned, possibly in combination with a paper mill.

The cleansed, nitrogen rich condensate, or at least a part of said condensate, may also be delivered to the traditional consumer stations.

Advantages

The inventive method prevents the emission of nitrogen oxides when burning nitrogen rich stripper gases and/or nitrogen rich methanol deriving from contaminated condensates. This is important primarily from an environmental aspect. When the emission of nitrogen oxides is taxed, the advantage is added that the pulp manufacturing will be cheaper to a certain extent.

Another advantage afforded by the invention is that the cleansed, nitrogen rich condensate is available for use for some specifically useful purpose, for instance in the biological purification of sewage or effluents where a nitrogen deficiency exists.

Description of the drawing

Figure 1 is a flowsheet which illustrates the processing of condensate, that derives from an alkaline pulp manufacturing process, in accordance with the invention.

Best embodiment

A preferred embodiment of the inventive method will now be described with reference to Figure 1. Certain part stages of the method will be described in more detail in conjunction herewith. This description is followed by the description of a working example in which the inventive method has been simulated in a laboratory.

In the process diagram of Figure 1 a continuous digester 1 is shown. Lignocellulosic material, for instance

wood in chip form, and cooking liquor in the form of white liquor, possibly mixed with cooking waste liquor, are delivered to the digester. A high temperature, e.g. a temperature of up to 170°C, prevails in the digester, resulting in powerful overpressure. The wood is digested by the cooking liquor to produce cellulose pulp, under described conditions, said pulp leaving the digester 1 normally at its bottom or in the proximity thereof. Handling of the lignocellulosic material and subsequent handling of the cellulose pulp during the pulp manufacturing process has not been included in the diagram for space reasons and for clarity reasons. Cooking waste liquor under pressure is removed from the digester 1 subsequent to digestion of the wood chips, i.e. at a given digester location. This cooking waste liquor is conducted through conduit 2 to a first flash tank 3 and from there through conduit 4 to a second flash tank 5. The main function of the flash tanks 3 and 5 is to reduce the pressure of the cooking waste liquor in steps, from a pressure of about 0.7 MPa down to atmospheric pressure. A gaseous mixture of the aforedescribed kind departs as the pressure is lowered. The gas mixture is conducted from the flash tank 5 to a condenser 7, through conduit 6. In the condenser 7, which can also be called a heat exchanger, the hot gas mixture comes into contact with cold plate walls or cold tubes (the opposite side of the plate wall or the tube wall is normally cooled with comparatively cold water) and the gas mixture condenses to a liquid or condensate. Alternatively, the mentioned walls and tubes can be omitted and the cold water brought into direct contact with the gas mixture, so as to condense the gas. This condensate, designated here the digester house condensate, is conducted through conduit 8 to a collecting vessel or condensate tank 9. Similar kinds of gas mixtures are taken out at several places in the digester house (not shown in the Figure). One example of this is the presteaming vessel (not shown in the Figure) located upstream of the actual digester 1. Since gas mixtures are taken out at

several places in the digester house and are all caused to condense (and the condensate then passed to the condensate tank), the resultant condensate is designated here the digester house condensate.

Bleaching waste liquor, which meets the digested lignocellulosic material in contraflow, is normally delivered to the bottom of the digester 1. The major part of the used cooking liquor, i.e. the cooking waste liquor, is displaced from the continuous stream of cellulose pulp and out of the digester 1 by the bleaching waste liquor, and the resultant mixture of bleaching waste liquor and cooking waste liquor, designated black liquor and also thin liquor, is collected and passed to the evaporation plant 10. The dry solids content of the black liquor or thin liquor is increased in the evaporation plant 10 from about 18-20% to perhaps more than 70%. This is effected by heating the thin liquor indirectly with steam from the soda recovery boiler step by step, each step normally being referred to as an effect. There is obtained within each effect (or in direct connection with each effect) a condensate which is designated evaporation condensate. Condensates that vary from contaminated to highly contaminated condensates are obtained in the majority of said effects (or in direct connection with said effects). The condensate obtained with one of said effects or possibly two of said effects, is relatively clean and can be removed from the system and used for some suitable purpose in the highly comprehensive pulp manufacturing process, up to the finally bleached cellulose pulp.

For the sake of simplicity and also clarity, the diagrammatic illustration of Figure 1 shows solely the removal of a contaminated or impure gaseous mixture from the evaporation plant 10. This mixture is passed through a conduit 11 to a first condenser 12, and through a conduit 13 to a second condenser 14. The condensate that is formed in these two positions is conducted to the condensate tank 9

through a conduit 15. The described condensate is designated the evaporation condensate.

The division between digester house condensate and evaporation condensate differs from mill to mill, and may also vary to some extent over a period of time in one and the same mill. The condensate mixture in the tank 9 will normally have a temperature in the range of 50-60°C. Although the pH of the condensate mixture may vary, it is normally higher than 9, for instance about 9.5.

In accordance with the invention, an acid, such as sulphuric acid for instance, is delivered to the condensate mixture through the conduit 16. The amount of acid delivered will be sufficient for the condensate mixture to have a pH of 7.5 or lower. According to one preferred embodiment of the invention, the acid is delivered in an amount such that the condensate mixture will have a pH of 6.5 or lower.

The pH-adjusted condensate mixture is taken out at the bottom of the tank 9 and passed to the heat exchanger 18 through the conduit 17. The condensate is then passed to the stripper column 20, through the conduit 19. The temperature of the condensate at this stage will typically be 95-110°C. Water vapour, fresh steam and/or vapour taken from some position in the evaporation process is delivered to the bottom of the stripper column 20 through the conduit 21, for stripping (driving off) earlier mentioned chemicals in vapour form, these chemicals being considered as and constituting contaminants.

The temperature of the condensate furthest up in the stripper column 20 will typically be 100-120°C. The amount of, e.g., fresh steam delivered through the conduit 21 is designated the steam ratio and may typically reach 0.2, which implies a delivery of 40 tonnes of steam per hour with respect to an hourly delivery of 200 tonnes condensate through the conduit 19. The degree of stripping effected in the stripper column 20 is usually controlled by determining the content of chemical oxygen consuming substance = COD

(Chemical Oxygen Demand) in the incoming condensate at position 19 on the one hand and in the outgoing cleansed condensate at position 22 on the other hand. The mean value of nine measurements taken over a given time period showed that the COD of the ingoing condensate was 10756 mg/l and in the outgoing condensate was 498 mg/l. With respect to contaminants in the condensate measured as COD, this shows that about 95% were removed by the stripper treatment and the stripper treatment is normally controlled so that at least 90% of the contaminants will be removed from the contaminated condensate, measured as COD.

The condensate present at the bottom of the stripper column 20 and having been cleansed of the major part of said contaminants, and therefore designated clean condensate, is removed from the bottom of the column 20 and passed to the heat exchanger 18, through the conduit 22. This heat exchange process lowers the temperature of the outgoing, cleansed condensate to a typical temperature of 65-70°C, and the condensate is passed to an appropriate consumer station through the conduit 23. As a result of lowering the pH of the condensate entering the stripping process in accordance with the invention, the major part of the nitrogen originally present in the incoming condensate is retained in the outgoing, cleansed condensate.

Departing stripper gases are passed to the condenser 25, through the conduit 24. Part of the gases condense to form a condensate in this position, this condensate being returned to the stripper column 20 through the conduits 26 and 19. The substances that remain in a gaseous state leave the condenser 25 through its top and are passed to the methanol column 28, through the conduit 27. The amount of gas delivered to the methanol column 28 may correspond roughly to 2 tonnes per hour. Part of the gas entering the column 28 converts to a liquid state, meaning that a limit between a lower liquid phase and an upper gas

phase will be found somewhere in the column. The temperature at the top of the methanol column 28 will typically be 70-75°C. Water vapour, fresh steam and/or vapour from some other position in the evaporation process is delivered to the methanol column 28 through the conduit 29.

The amount of water vapour, steam, delivered may correspond to 500 kg per hour. Mainly gaseous methanol is expelled from the column 28 and passed to the partial condenser 31, through the conduit 30. Part of the gas condenses in the condenser and the resultant condensate is passed back to the methanol column 28, through the conduit 32. A certain amount of liquid is withdrawn from the bottom of the methanol column 28 and passed to a decanter or turpentine separator 34, through the conduit 33. As the name implies, turpentine is extracted in the decanter and passed to the vessel or tank 40, through the conduit 35. Liquid that remains in the decanter 34 is passed back to the stripper column 20, via the conduit 36.

The partial condenser 31 will typically have a temperature of 63-67°C. Remaining substances in gas form are passed to a so called methanol condenser 38 through the conduit 37. This methanol condenser typically has a temperature of 30-35°C, therewith causing substantially all gaseous methanol to condense to a liquid form, this liquid being passed to the methanol tank 40 through the conduit 39.

Those gases that are unable to condense at the described temperature are taken from the top of the methanol condenser 38 and passed through a conduit 41 to a incinerator (gas boiler) 42, where the odorous sulphur containing substances constituting the major part of said gas mixture are combusted and destroyed. When processing the contaminated or impure condensate in accordance with the invention, the nitrogen content (mainly in the form of ammonia) of said gas mixture is very small and possibly even down to a zero level.

The liquid passed to the methanol tank 40 through the conduit 39 will normally consist of more than 80%

methanol. The liquid methanol is passed to the incinerator 42 through the conduits 43 and 44 and serves as fuel in the incinerator for the combustion and destruction of the aforesaid odorous gases. Normally, the amount of methanol formed is greater than the amount required as fuel in the incinerator 42, and the excess methanol is delivered to the lime kiln 46 through the conduits 43 and 45, and there used as a backup fuel in the conversion of lime sludge, to burnt lime. The mentioned methanol is also essentially free of nitrogen.

Because the odorous gas mixture and the methanol are both essentially free of nitrogen, the combustion of these substances will at most result in only the very slight formation and emission of environmentally hazardous nitrogen oxides through the exhaust gases in respective positions.

The process diagram shown in Figure 1 illustrates a conventional method of dealing with the stripper gases, i.e. from position 24 and forwards in the process. Although not illustrated and described in detail in this document, alternative methods of dealing with said stripper gases are available, and the inventive method can also be applied in conjunction with these processes.

In the mentioned process diagram, the digester house condensate arrives from a continuous digester house. However, it will be understood that condensate generated in batchwise cooking processes can also be treated in accordance with the invention.

Example 1

A certain quantity of contaminated or impure condensate was taken from a sulphate pulp mill, said condensate consisting of a mixture of digester house condensate and evaporation condensate. The condensate was divided into subquantities of 100 ml in a laboratory.

The original condensate had a pH of 9.57 and different quantities of 0.1 molar sulphuric acid were added

to said subquantities, leading to a test series with stepwise falling pH values.

Respective samples were subjected to a simulated stripping process in the laboratory, by reflux boiling in a round flask equipped with cooling means, for five minutes. When the sample had cooled, its pH value and content of dissolved ammonia plus ammonium ions was measured. The ammonia plus ammonium content of the original condensate was also determined.

The ammonia content plus ammonium content of the samples and of the original condensate were determined with Dr. LANGE Kyvett test LCK 303, edition 95/07.

The measured pH values as well as ammonia and ammonium contents and nitrogen contents respecitively (recalculated) of the test series are listed in the following table.

Test No.	*pH*	*NH_3 + NH_4^+ mg/l*	*N mg/l*
1 (no acid addition)	9.57	73.6	57.2
2	9.24	112.0	87.0
3	9.13	122.0	94.7
4	8.98	140.0	109.0
5	8.85	152.0	118.0
6	8.66	161.0	125.0
7	8.48	173.0	134.0
8	7.78	212.0	165.0
9	6.98	221.0	172.0
10	6.61	236.0	183.0
11	5.48	254.0	197.0

The $NH_3+NH_4^+$ concentration of the original condensate was 255.0 g/l, and the N concentration was 198.0 mg/l.

As will be evident from the table, 80% of the nitrogen remained in the condensate subsequent to the simulated condensate stripping process at a pH of immediately below 8. At a pH of 5.5, substantially all nitrogen remained in the condensate subsequent to the simulated condensate stripping process.

It is assumed that all nitrogen in the different samples that departed from the samples and the round flask left in the form of gaseous ammonia.

The tests indicate that the major part of the nitrogen content of the original condensate can be retained in the condensate subjected to a stripping process also on a technical scale, when said process is carried out in accordance with the invention.

CLAIMS

1. A method of processing condensate deriving from the manufacture of cellulose pulp by alkaline digestion, **characterized** by adding acid to the condensate prior to a stripper treatment process so as to lower the pH of the condensate to ≤7.5 leading to retainment of the major part of the original nitrogen content of said condensate; and handling stripper gases generated by the stripper treatment in a conventional manner and passing cleansed, nitrogen rich condensate to a consumer station.

2. A method according to Claim 1, **characterized** by adding acid to said condensate prior to said stripper treatment such as to lower the pH of the condensate to ≤6.5.

3. A method according to Claims 1-2, **characterized** in that the condensate consists exclusively of evaporation condensate.

4. A method according to Claims 1-2, **characterized** in that the condensate consists exclusively of digester house condensate.

5. A method according to Claims 1-2, **characterized** in that the condensate consists of a mixture of evaporation condensate and digester house condensate.

6. A method according to Claims 3-5, **characterized** in that the acid is delivered individually to each respective condensate streams, or is delivered at a position or point in which said two streams are mixed together, or is delivered at a position which is located downstream of the mixing point but upstream of the stripper treatment.

7. A method according to Claims 1-6, **characterized** by passing all or a part of the cleansed, nitrogen rich condensate to a biological sewage or effluent purification system.

8. A method according to Claims 1-6, **characterized** by using all or a part of the cleansed, nitrogen rich condensate as washing liquor in the manufacture of cellulose pulp and/or in the chemical recovery system.

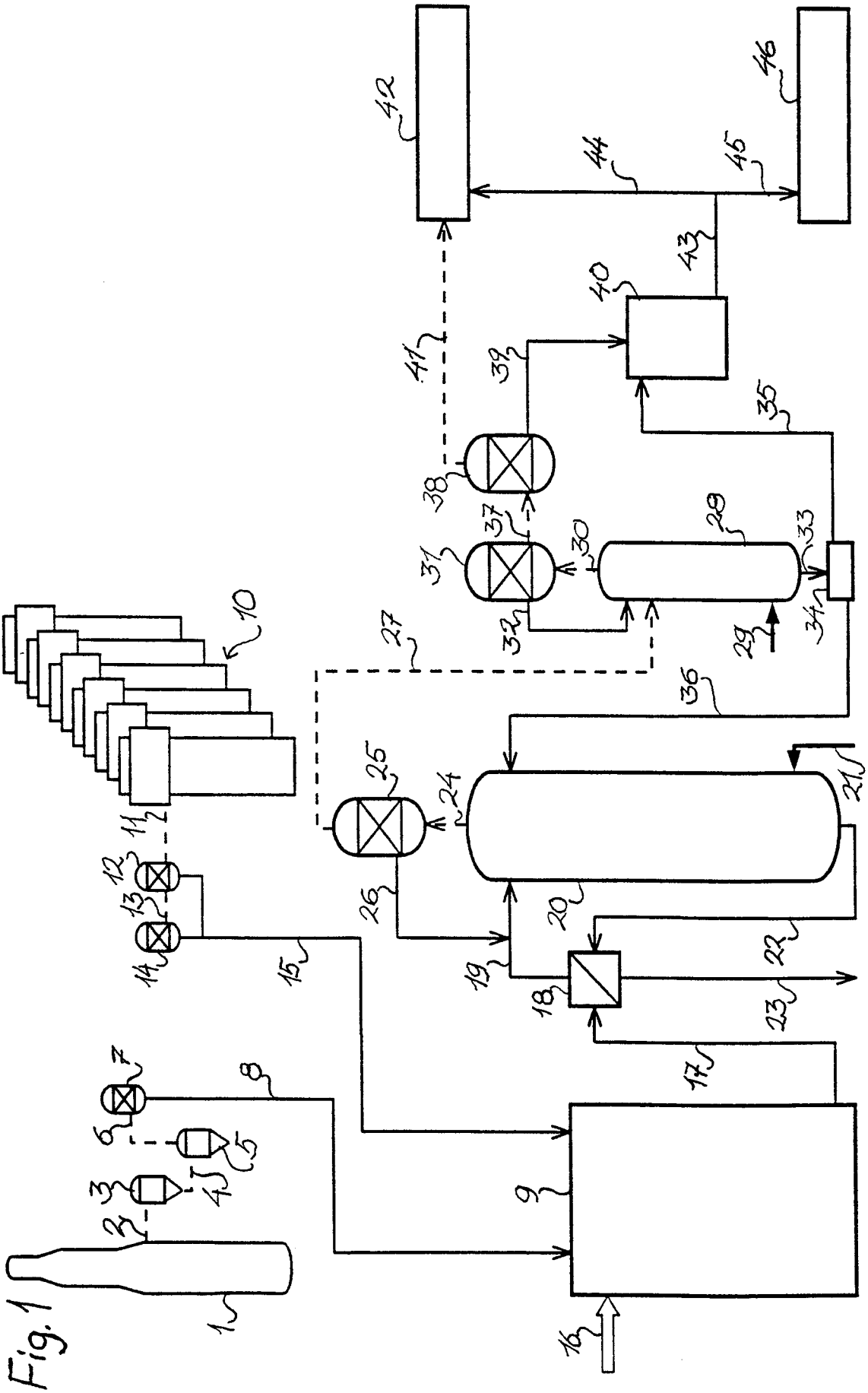
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Fig. 1

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 99/00068

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: D21C 11/10, D21C 11/00
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: D21C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 9623566 A1 (KVAERNER PULPING AB), 8 August 1996 (08.08.96), claims 1,4	1-8
A	EP 0521308 A1 (KRUPP KOPPERS GMBH), 7 January 1993 (07.01.93), abstract	1-8
A	FR 2444645 A1 (LA CELLULOSE DU PIN ET AL), 18 July 1980 (18.07.80), claim 1	1-8
A	EP 0038686 A2 (HELIX TECHNOLOGY CORPORATION), 28 October 1981 (28.10.81), abstract	1-8

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" erlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
10 May 1999	18 -05- 1999
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Helena Hemphälä Telephone No. +46 8 782 25 00

INTERNATIONAL SEARCH REPORT

Information on patent family members

07/04/99

International application No.
PCT/SE 99/00068

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 9623566 A1	08/08/96	AU 2270695 A	30/10/95
		FI 973173 A	31/07/97
		SE 503793 C	09/09/96
		SE 9500376 A	02/08/96
EP 0521308 A1	07/01/93	CZ 281998 B	16/04/97
		DE 4135763 A	07/01/93
		DE 59200123 D	00/00/00
		HU 66417 A	28/11/94
		HU 210507 B	28/04/95
		PL 168766 B	30/04/96
		SK 206192 A	10/11/93
FR 2444645 A1	18/07/80	CA 1148674 A	21/06/83
		FI 793950 A	19/06/80
		SE 7910342 A	19/06/80
EP 0038686 A2	28/10/81	AT 7762 E,T	15/06/84
		AU 550666 B	27/03/86
		AU 7170881 A	10/11/81
		CA 1158838 A	20/12/83
		IN 153700 A	11/08/84
		JP 57500646 T	15/04/82
		US 4293322 A	06/10/81
		US 4383841 A	17/05/83
		US 4462814 A	31/07/84
		WO 8103063 A	29/10/81

LIITE III

Muiden työryhmien kuulumiset

YTR
Käynnissä olevat projektit

SUOMEN SOODAKATTILAYHDISTYS
FINNISH RECOVERY BOILER COMMITTEE

BAT-dokumentin kommentointi

- Dokumenttiluonnos on poistettu IPPC:n sivuilta, mikä tarkoittanee että dokumenttitekstiin on tulossa vielä muutoksia.
- Helmikuussa 2013 pidetään teknisen työryhmän (TWG, technical working group) kokous
- Julkaisuaikataulu?

POPE

- Hiukkasmittauksia tehty Kymillä soodakattilasta (pesurin jälkeen) vko 34 -> vaikeuksia saada tarpeeksi hiukkasia impaktoriin
- SK uusinta mittaukset vko48 sekä pesurin että sähkösuodattimen jälkeen
- Meesauunimittaus vko 49
- Johtoryhmän kokous oli 20.11, pöytäkirja
- Seuraava kokous 13.6.2013 Kuopio

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Hajukaasusuosituksen päivitys

- Päivitystyö melkein valmis, teksti läpikäyty kaksi kertaa
  - Metanolin/tärpätin poltto-kappale vielä tarkistettava
- Edellinen kokous 10.12.2012
- Vielä yksi kokous 23.1.2012 Imatralla, Konemestaripäivien yhteydessä
- Suosituksen laajentaminen keräilyyn?

YTR
Projektiehdotukset

SUOMEN SOODAKATTILAYHDISTYS
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Ammoniakin talteenotto stripperin lauhteista

- Projektissa "Ammonia Formation and Recovery in a Kraft Pulp Mill and Fate of Biosludge Nitrogen" todettiin että suurin osa talteenotettavasta ammoniakista on likaislauhteessa
  - The ammonia found in the dirty condensates would represent 75% of this ammonia while the other 25% of this would be found in the vent gases from recausticizing. Since the ammonia in white liquor is ultimately found in the dirty condensates from pulping and evaporation, its flow from recausticizing is not included in the potential nitrogen recoverable from recausticizing. One challenge of recovering the NH3 from the dirty condensates would be to separate it apart from the MeOH.

Ammoniakin talteenotto stripperin lauhteista 2/2

- Ammoniakki saataisiin pysymään lauhteessa ammoniumina stripperin pH:ta muuttamalla happamaksi rikkihapolla, pH alle 7 voisi riittää
- Menetelmä patentoitu: http://patentscope.wipo.int/search/en/detail.jsf?docId=WO1999037853&recNum=1&maxRec=&office=&prevFilter=&sortOption=&queryString=&tab=PCT+Biblio
- Pohdittavaa:
  - mitä vaikutuksia stripperin ajolla on happamana, korroosio, muuttuuko kiehuminen, saostuuko jotain?
  - stripatun lauhteen käyttökohteet, mihin ammoniakki päätyy

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Sähkösuodintuhkan hyötykäyttö 1/2

- Sellutehtaan ylijäämäisen rikkitaseen hallitsemiseksi on tapana liuottaa lentotuhkaa ja viemäröidä se jäteveden mukana. Ympäristölupien uusinnan yhteydessä tehtaille voi tulla rajoituksia tähän käytäntöön
- Tuhkan vieminen kaatopaikalle ei ole sallittua koska tuhka ylittää kaatopaikkajätteelle määritellyn liukoisuusrajan, joten keinot päästä eroon tuhkasta on liuotus tai hyötykäyttö.

Sähkösuodintuhkan hyötykäyttö 2/2

- Vaihtoehtoja hyötykäytölle:
  - Puhtaan natriumsulfaatin valmistus selkeytys/kiteytyksellä (yhdistyksen aikaisempi tutkimus)
  - NaOH ja rikkihapon valmistus natriumsulfaatista elektrohydrolyysillä (tutkittu 1980-luvulla, kannattavuus riippuu NaOH markkinahinnasta)
- Diplomityö?
- Yhdistyksen projekteissa vuosina 2003-2009 tutkittiin sähkösuodintuhkan puhdistusta ja käsittelyä natriumsulfaatin hyötykäyttöä ajatellen. Lisäksi selvitettiin mahdollisia natriumsulfaatin käyttökohteita sekä tuotteen kuljetusta

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Hiukkaskokojakaumat ja pölyemissiot

- Selvitettäisiin jakaumat ja kemiallinen koostumus ennen ja jälkeen sähkösuodattimen, tämän päivän tilanne
  - EPA:lla kaksi mittaustapaa sähkösuodattimelle
  - Vaatimukset raskasmetalleille / BAT rajat
  - Höngissä paljon PM1 hiukkasia
- Aikaisempi tutkimus julkaistu artikkelina Environmental & Science Technology lehden numerossa 2/2006.
  - http://pubs.acs.org/doi/abs/10.1021/es0503027

Sellutehtaan dioksiinipäästöt

- Jatkotutkimuksena selvitys dioksiinipäästöihin vaikuttavista tekijöistä, ajoparametrit, lämpötila
- Aikaisempia tutkimuksia on tehnyt Vic Uloth, Paprican.
- Dioksiinipitoisuuden määrityksen hinta riippuu detektiorajasta, mitä tarkempi sen kalliimpi. Näytteet lähetettäisiin nimettöminä.
- Sihteeri tiedustelee jäsenyhdyshenkilöiltä nähdääkö tälläinen projekti tarpeelliseksi ja onko mitattu, jos on, onko tuloksia saatavilla.

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IE-direktiivin vaikutukset soodakattilan päästöarvoihin

- Soodakattilat/meesauunit ulkona LCP-määräyksistä
  - IE-direktiivissä kiinteät päästörajat, joista ei voi poiketa
  - Soodakattila erottamaton osa tehdasta, EU tuomioistuimen päätös olemassa -> ei voi olla jätteenpolttokattila
  - LCP-laitoksille polttoaine tuodaan ulkopuolelta -> soodakattila ei ole LCP-laitos
- Komissio tarkastelee kuitenkin parhaan käytettävissä olevan tekniikan pohjalta, onko tarpeen vahvistaa EU-laajuisia päästöraja-arvoja muun muassa soodakattiloille
- Komissio antaa 31.12.2013 mennessä kyseisen tarkastelun tuloksista kertomuksen parlamentille ja neuvostolle sekä tarvittaessa säädösehdotuksen

NOx hallintatekniikat

- Kattilan ajotapa
- NOx:n mittaaminen oikein
  - Jatkuvatoiminen vai kertamittaus
  - Näytteenkäsittely
  - Savukaasumäärän mittaaminen
  - Yhteistyö Ruotsin kanssa

Altistuminen tuhkalle sellutehtaalla ja tuhkan käsittelyssä:

- Työterveyslaitos tehnyt tutkimuksen biopolttolaitoksen tuhkien koostumuksesta ja työhygieenisistä riskitekijöistä huolto- ja korjaustöissä
- Mitattujen epäpuhtauksien ja niiden pitoisuuksien perusteella arvioitiin käytössä olevan suojautumistason riittävyyttä
- Raportti:

Soodakattilan raskasmetallipäästöt ilmaan

- ÅA diplomityö (Lipeätyöryhmä) raskasmetallitaseista rajautui sähkösuodattimeen, mutta ainakin elohopea läpäisee sähkösuodattimen
- Euroopassa sijaitsevien tehtaiden päästöjä voi tarkastella E-PRTR-rekisteristä. http://prtr.ec.europa.eu/

SUOMEN SOODAKATTILAYHDISTYS
FINNISH RECOVERY BOILER COMMITTEE

Työryhmän kommentit

- Ulkoasultaan moitteeton
- Kattava yleisosuus sekä laboratorio- että tehdastutkimuksia
- Tuloksia on verrattu aiempiin tutkimuksiin ja teoriaan, lähdeviitteitä on kattava määrä
- Käytetyt menetelmät on tarkasti raportoitu ja lisäksi tulosten luotettavuutta arvioitu
- Kokeellisessa osassa on perehdytty analysointimenetelmiin ja kehitetty menetelmä suovan mustalipeäpitoisuuden määrittämiseksi
- Hyviä ehdotuksia jatkotutkimuksista
- Asetetut tavoitteet saavutettiin eli tuloksilla saatiin aikaan konkreettinen parannus tehtaan mäntyöljyn tuotantoon

SKYREC

SUOMEN SOODAKATTILAYHDISTYS
FINNISH RECOVERY BOILER COMMITTEE

Things to be done

- Last meeting 19.12.2012
  - Accepting final report of furnace material tests, VTT
  - Results from project Probe Study of Corrosion in the Economizers of a Kraft Recovery Boiler, ÅA
- Accepting final report

Probe Study of Corrosion in the Economizers, ÅA

- Two probes in Rauma mill
  - Around 700 hours for the first probe before shutdown 5th October. Probes temperature is 90 C.
  - Second probe will there during shutdown and pulled out before chemical purification
- Before start-up chemical purification of the boiler tubes
  - Acid dew point measurement during that time

Timeline – Probe Studies

Date In	Date Out	Probe Temperature	Comments
31.8.2012	31.8.2012	80 °C	2h probe test
31.8.2012	31.8.2012	75 °C	2h probe test
31.8.2012	4.10.2012	90 °C	811h probe test, probe pulled before water wash
31.8.2012	17.10.2012	90 °C	1124h probe test, probe pulled after water wash, but before oil firing
19.10.2012	19.10.2012	90 °C	2h probe test during oil firing

Corrosion results

Probe Time	Probe Temperature	Ring Weight Loss (mg)	Avg. Corrosion Layer Thickness (µm)	Calculated Avg. Corrosion (mm/yr)
2 h	80 °C	0	0	
2 h	75 °C	0	0	
811 h	90 °C	22	2*	0.02
1124 h	90 °C	72	6**	0.05
2 h	90 °C (oil)	0	0	

*Pit corrosion, pits ~10 µm deep
**Pit corrosion, pits ~20 µm deep

Muiden työryhmien toiminta

Käynnissä olevat projektit

ATR: Ohje UPS-järjestelmän periaatteeksi

- Projektia varten perustettu työryhmä jättänyt loppuraportin:
  - Sähkön syötölle tulisi mahdollisuuksien mukaan tarjota useampi toisistaan täysin riippumaton kulkutie. Yksittäisiä yhteisiä osia tulisi välttää (automaattinen syötönvaihto, yhteinen UPS kisko)
  - UPS-laitteiden lisääminen parantaa lähinnä sähkönsyötön toimintavarmuutta katkostilanteessa. Varsinkin kun huomioidaan UPS laitteiden taipumus vikaantua juurikin tarvetilanteessa
  - Oleellista on kiinnittää huomiota myös normaalitilanteen sähkönsyötön varmuuteen kriittisten laitteiden ja järjestelmien osalta
- ATR pohtii projektin jatkoa -> Ohjeen teko

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KTR: Aktiivihiilen mitoituksen varmistus ja optimointi sekä TOC-reduktion varmistaminen, JPanalysis/OY

- Tarkoitus varmistaa suodattimen mitoitus koeajoilla eri virtaamilla
- Selvitetään aktiivihiilisuodattimen kustannukset ja toimittajat
- Lisäksi vertaillaan kahden TOC-laitteiston antamia tuloksia.
- Työ viivästynyt TOC-laitteen hajoamisen takia -> työt päästy vasta aloittamaan

KTR: Soodakattilanmateriaalit ja tarkastukset (Suojaussuosituksen päivitys)

- VTT päivittänyt osan 2 (Soodakattilan pinnoitteet)
- Pinnoitettavia kohteita ovat yleensä
  - ”mustan” ja kompound-putken raja, jossa pinnoitteella estetään galvaanista korroosiota
  - tulistinputkien mutkat, joissa pyritään estämään hapettumisen ja korroosion kautta tapahtuvaa ohenemista
  - paikalliset kompound-putkien säröytymien korjaus
- Tartunta tärkeää
- Korjaaminen hankalaa/mahdotonta

SUOMEN SOODAKATTILAYHDISTYS
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LTR: Syöttövesipumppujen säätö, LUT

- Työssä tehdään esiselvitys kolmen SKY:n valitseman soodakattilan syöttövesipumppauksen mahdollisuuksista säästää sähköä toteuttamalla pumppauksen säätö uudella tavalla
  - Taustalla on ABB:n rahoittama väitöskirja jossa on tutkittu taajuusmuuttajapumppujen energiataloudellista ajotapaa
  - Samalla mietitään miten suurella syöttövesisäiliöllä kukin soodakattila pärjäisi.
  - Lisäksi mietitään mikä olisi energiataloudellisen syöttövesipumppu konfiguraatio
- Tutkittavat kattilat: Veracel (data puuttuu), Fray Bentos (data puuttuu), Joutseno (data puuttuu)

Konemestaripäivät 23-24.1.2013

- Konemestaripäivät järjestetään 23.–24.1.2013 Imatran Valtionhotellissa ja tehdasvierailu Metsä Fibren Joutsenon tehtaalla.
- Ajankohtaa jouduttiin aikaistamaan viikolla tehdasvierailun vuoksi
- Ohjelma:
  - Henkilöturvallisuuden parantaminen soodakattilalaitoksessa RFID avulla, http://www.visi.fi/
  - Soodakattila- ja turbiinivauriot, Thomas Åström (Pohjola)

SUOMEN SOODAKATTILAYHDISTYS
FINNISH RECOVERY BOILER COMMITTEE

SKY 50v ja ICRC 2014 9.6-13.6.2014

- ICRC-seminaari 9.-13.6.2014 Tampere-talossa
- 50v-toiminkunta on perustettu miettimään juhlapäivän ohjelmaa/ luennoitsijoita
  - Timo-Pekka Veijonen, Keijo Salmenoja, Mikko Hupa, Klaus Niemelä, Esa Vakkilainen
- Alustavat nettisivut (ei vielä julkaistu): http://www.soodakattilayhdistys.fi/secure/ICRC/ICRC_index.html

Vuosikokous 2013

- Helsingissä 18.4.2013
- Saman konseptin mukaan viime kerralla (itse kokous iltapäivällä ja iltaohjelma sen jälkeen)
- Sihteeri kartoittaa mahdollisia kokouspaikkoja (musiikkiteattereita).

SUOMEN SOODAKATTILAYHDISTYS
FINNISH RECOVERY BOILER COMMITTEE

Projektiehdotukset